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PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION under 37 CFR 1.53(b)(2)

	Docket Number	P-122314.02 (PRO)	Type a plus sign (+) inside this box ->	+

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Title of the Invention

CONTROLLED LOW STRENGTH FLOWABLE FILL COMPOSITION

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ENCLOSED APPLICATION PARTS *(check all that apply)*

[X] Specification/claim: Number of Pages: 10	[ ] Combined Declaration/Power of Attorney
[X] Drawing(s): Number of Sheets: 1	[ ]

METHOD OF PAYMENT *(check one)*

	PROVISIONAL FILING FEE AMOUNT ($)	$80.00
[x] A check or money order is enclosed to cover the provisional filing fees [x] The Commissioner is hereby authorized to charge any deficiency in fees and credit Deposit Account Number: 07-2400		

[x] Applicant qualifies as a small entity under 37 CFR 1.9(c).

This invention was made by an agency of the United States Government or under contract with an agency of the United States Government.

[x] No.
[ ] Yes, the name of the U.S. Government agency and the Government contract number are:
____

Respectfully submitted,

SIGNATURE ______________ Date: March ~~7~~ 10, 2003

Thomas E. Sisson, Reg. No. 29,348

[ ] Additional inventors are being named on separately numbered sheets attached hereto.

CERTIFICATE OF EXPRESS MAILING

I hereby certify that this paper (along with any paper referred to as being attached or enclosed) is being deposited on the date shown below with the United States Postal Service, as Express Mail Post Office to Addressee (37 CFR 1.10), Mailing Label No. EL692001553US, addressed to the Commissioner for Patents and Trademarks, Washington, D.C. 20231.

Date: 3-10-03

Bianca Grossweiler

Bianca Grossweiler

Title: CONTROLLED LOW STRENGTH FLOWABLE FILL COMPOSITION

Inventors: Jerry Setliff, Scott F. Timmons, and Clinton W. Pike

BACKGROUND OF THE INVENTION

This invention relates to Controlled Low-Strength Mixtures (CLSMs), or flowable back-fills. This class of materials has utility as pipe bedding materials where they are used to both protect the pipe from external agents and internal loads. They have also been used as an erosion barrier in embankments and as a mine fill material. CLSMs typically have strengths of less than 2000 psi and, in cases where removal is contemplated, less than 200 psi for ease of removal. The material should be initially in the form of an easily pumpable, self-leveling slurry. Rapid early strength development (approximately 50-70 psi) is a desirable property and is currently not obtainable with commercial products without the penalty of high strength development at later stages. U.S. Patent No. 5,106,422 discloses Class C Fly ash in a rapid setting flowable backfill composition and method for its use.

However, such existing compositions are based upon the use of either Portland cement or Class C fly ash used individually or in combination as the hydraulic cement component of the CLSM system. Typically these cementitious materials are used at less than 5% by weight in the case of Portland cement or as much as 50% in the case of Class C fly ash with the remainder being some form of aggregate, usually fine sand or soil from the spoil with small amounts of additional rock and gravel or Class F fly ash. Cement-based materials can take days to hydrate, cure, and achieve even a

modest strength of 50 psi which is typically the minimum strength required for a man to walk upon the surface of the bedding material and represents the minimum safe time before the cover fill may be placed. Class C fly ash based systems may take as long as four hours to hydrate, cure, and achieve this strength. In many cases, locally available Class C fly ash is not desirable for use in these types of product due to slow hydration, cure, and set times and low strengths. Strength may be compensated for by the use of additional Class C fly ash but the cost of the additional fly ash may result in cost prohibitive products.

Thus, the system of the present invention minimizes the down time before cover fill may be placed and represents a significant savings of both time and money for the user. Furthermore, the present inventive composition and method allow for control variability in strength and hydration, cure, and set times of a CLSM system utilizing Class C fly which results in a Class C-based flowable fill capable of competing in markets previously inaccessible.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a graphic representation of the effect of set time of Class C mortars with lime.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Class C fly ash as defined in ASTM C 618 is a coal combustion product that meets particular size requirements and mineralogical specifications. A typical chemical composition for this class of fly ash is as follows:

	Percent by Weight
Silicon dioxide (SiO_2) plus aluminum oxide (Al_2O_3) plus iron oxide (Fe_2O_3), min.	50.0
Sulfur trioxide (SO_3), max.	5.0
Moisture content, max.	3.0
Loss on ignition, max.	6.0

This is a rather broad description for this class of material, and significant variability may exist for materials conforming to this requirement. The variability manifests itself as differences in hydration and set time and strength between several samples of Class C fly ash either from the same or different sources. It has been found that a major factor contributing to variability is the amount of available calcium present in the sample. Additionally, soluble iron content contributes to slow setting times. Furthermore, the addition of small amounts of calcium to Class C fly ash has no deleterious effects upon flowable fill and can accelerate the rate of hydration and cure while minimizing the differences in set time and strength of flowable fill mixtures containing Class C fly ash.

Where soluble iron is present in sufficient quantity and extra calcium alone is inadequate to accelerate the rate of hydration, iron chelating compounds may be added, even in very small amounts, to offset the soluble iron effect. The iron chelating compound may include:

sodium chloride, sodium thiosulfate, triethanolamine, diethanolamine, polyethyleneimine, amino-substituted acrylic monomers or polymers, morpholine and substituted morpholine compounds, urea, guanidine salts, pyrole and pyrole compounds, polyvinylpyrole, imidizole compounds, pyrazoles, pyridine and pyridine compounds (especially ortho alkoxy-

substituted pyridines), amino phenol (especially ortho amino phenol), amino cresol, ortho anisidine, amine acetate surfactants (such as Armac HT and Armac 18D-40 from Akzo Nobel Chemicals), amine oxide surfactants (such as Ammonyx series of surfactants from Stepan Company, Schercamox series of surfactants from Scher Chemicals, Foamox series of surfactants from Alzo, Inc., Chemoxide series of surfactants from Chemron Corp.,), amine surfactants (such as the Armeen and Redicote series of surfactants from Akzo Nobel Chemicals, the Incromine series of surfactants from Croda, Inc., the Tealan series of surfactants from R.I.T.A. Corp.), and mercapto surfactants (such as Burco TME from Burlington Chemicals).

The iron chelating compound may be in quantities in the range of 0.01% or 5.0% by weight. Effective results have been obtained and reasonably should be obtained from chelting agents or compounds selected from the group consisting of an alkanolamine, a polymer of ethyleneimine, a block copolymer containing polyethyleneimine segments, an amino-substituted polymer of acrylic acid, the salt of an amino-substituted polymer of acrylic acid, a carboxyated amine compound, a salt of a carboxyated amine compound, ethylenediaminetetraacetic acid and salts thereof; nitrilotriacetic acid and salts thereof, an amine substituted surfactant, an amine oxide substituted surfactant, and a guanidine salt.

The following examples illustrate the nature of the present invention. Set times were determined when a 0.25" diameter penetrometer needle provided a reading of 200 psi on insertion to a depth of 1.0".

Example 1

Coal Fly ash from Deeley Power Plant, San Antonio, Texas, as obtained and used as received. 50 grams of Class C Fly ash, 250 grams ASTM C 33 graded washed silica sand (Espey Sand, San Antonio, Texas) and 35 mL deionized water were mixed for 1 minute and poured into a 2" cube mold. The set time was determined to be 62 minutes as shown in Table 1 below.

Examples 2-8

Coal Fly ash from Deeley Power Plant, San Antonio, Texas, was obtained and used as received. 50 grams of Class C Fly ash, 250 grams ASTM C 33 graded washed silica sand (Espey Sand, San Antonio, Texas), varying amounts of type S hydrated lime and 35 mL deionized water were mixed for 1 minute and poured into a 2" cube mold. The set time for these examples are shown in Table 1 for the varying amounts of lime.

Examples 9-13

Coal Fly ash from Scherer Power Plant, Atlanta, Georgia, was obtained and used as received. 50 grams of Class C Fly ash, 250 grams ASTM C 33 graded washed silica sand (Espey Sand, San Antonio, Texas), varying amounts of type S hydrated lime and 35 mL deionized water were mixed for 1 minute and poured into a 2" cube mold. The set times for these examples are shown in Table 1 for the varying amounts of lime.

A graphic representation of the effect of set time of these Class C motars with lime of varying amounts is shown in Fig. 1.

Table 1. Set times of Class C Fly ash motar cubes containing varying amounts of type S lime.

Lime (grams)	Example Number	Set Time (minutes) Deeley	Example Number	Set Time (minutes) Scherer
0.00	1	62	9	348
0.07	2	51	-	-
0.13	3	26	-	-
0.25	4	12	10	303
0.50	5	10	11	71
0.75	6	8	12	37
1.00	7	12	13	76
1.50	8	9		

Example 14

Ingredient	Amount (g)
Concrete Sand	0
Type C Fly Ash	100
Hydrated Lime	.003
Triethanolamine	.04
Water	25

The dry ingredients were mixed together and the water was added with mixing until a smooth, pourable consistency was obtained. The set time was 17 minutes.

Example 15

Ingredient	Amount (g)
Concrete Sand	200
Type C Fly Ash	100
Hydrated Lime	.3
Triethanolamine	.48
Water	39

The dry ingredients were mixed together and the water was added with mixing until a pourable consistency was obtained. The set time was 17 minutes.

Example 16

Ingredient	Amount (g)
Concrete Sand	250
Type C Fly Ash	50
Hydrated Lime	7.5
Triethanolamine	.1
Water	40

The dry ingredients were mixed together briefly and the water and triethanolamine added with continued mixing. The set time was 23 minutes.

CLAIMS:

1. A rapid setting, controlled low strength composition of Class C fly ash comprising hydrated lime in the amount of 0.1% to 15% by weight and an iron chelating compound in the amount of from 0.01% to 5% by weight sufficient to accelerate the hydration and set time of said fly ash.

2. A rapid setting, controlled low strength composition of Class C fly ash comprising hydrated lime in the amount of 0.1% to 15% by weight of fly ash and an iron chelating compound in the amount of from 0.01% to 5% by weight sufficient to accelerate the hydration and set time of said fly ash, and a filler material in the amount of 1:10 to 10:1 parts by weight.

3. A method by which the hydration and set time of a cementitious mixture containing Class C fly ash is accelerated comprising the step of adding hydrated lime in the amount of 0.1% to 15% by weight of and an iron chelating compound in the amount of from 0.01% to 5% by weight cementitious material to said cementitious mixture.

4. A rapid setting, controlled low strength composition of Class C fly ash comprising a calcium source in the amount of 0.1% to 15% by weight and an iron chelating compound in the amount of from 0.01% to 5% by weight sufficient to accelerate the hydration and set time of said fly ash.

5. The composition of claim 4 wherein said source is quicklime.

6. The composition of claim 4 wherein said calcium source is selected from the group consisting of calcium nitrate, calcium nitrite, calcium formate, calcium acetate, calcium proprionate, calcium lignosulfonate, calcium oxide, calcium hydroxide, calcium hypochlorite, anhydrous calcium sulfate, calcium sulfate dihydrate, and calcium sulfate hemihydrate.

7. The composition of claim 4 wherein said calcium source is a circulating fluidized bed coal ash containing free lime in the amount of 0.25% to 70% by weight of Class C fly ash.

8. The composition of claim 2 wherein said filler material is selected from the group consisting of Class F fly ash, silica sand, dolomitic calcium carbonate sand, limestone sand, expanded perlite, expanded styrofoam, bottom ash, slag, foundry sand, expanded shale, clay, ground granite sand, pumice and gravel.

9. The composition of claim 4 wherein said iron chelating compound is selected from the group consisting of an alkanolamine, a polymer of ethyleneimine, a block copolymer containing polyethyleneimine segments, an amino-substituted polymer of acrylic acid, the salt of an amino-substituted polymer of acrylic acid, a carboxyated amine compound, a salt of a carboxyated amine compound, ethylenediaminetetraacetic acid and salts thereof; nitrilotriacetic acid and salts theeof, an amine substituted surfactant, an amine oxide substituted surfactant, and a guanidine salt.

ABSTRACT OF THE DISCLOSURE

A rapid setting, controlled low strength composition of Class C fly ash is provided having a quantity of hydrated lime and an iron chelating compound in an amount sufficient to accelerate the hydration and set time of the fly ash. In some examples, a filler material is added. A method for acceleration of the hydration and set time of a cementitious mixture is provided wherein hydrated lime is added to the cementitious mixture in an amount in the range of 0.1% to 15% by weight and an iron chelating compound in an amount in the range of 0.01% to 5.0% by weight of the cementitious material. Further, a calcium source and an iron chelating compound may be added to a Class C fly ash to accelerate the hydration and set time of the ash.

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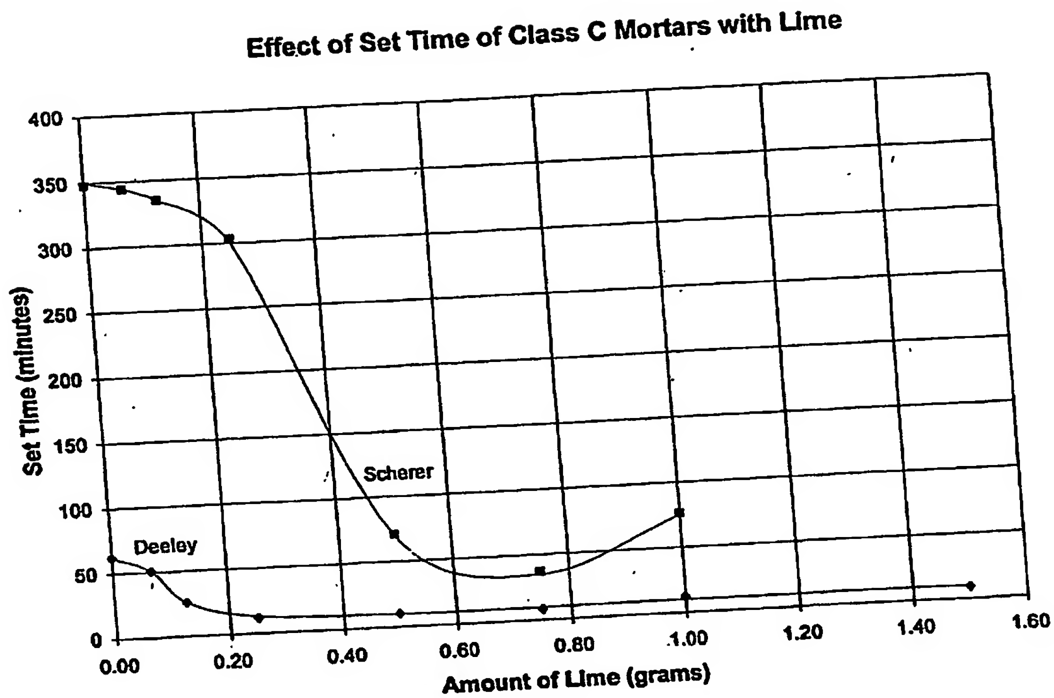

FIG 1.

[redacted]